EFFECT OF COMPACTION PRESSURE ON FINAL PROPERTIES OF MULTIWALL CARBON NANOTUBES (MWCNTs) REINFORCED ALUMINUM (Al) COMPOSITE

By

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CERTIFICATION OF APPROVAL

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A project dissertation submitted to the
Mechanical Engineering Programme
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(Mechanical Engineering)

Approved:

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December 2010

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

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Nurul Rawaida Ain Burhani

ABSTRACT

In aerospace and automotive industry specifically, the challenge is to fabricate material which have high strength to weight ratio composite. Multiwall Carbon nanotube (MWCNTs) reinforced Aluminum (Al) composites usage grows at an impressive pace due to the positives results in increasing hardness, tensile strength and Young's Modulus with low density. When the hardness and tensile strength obtained much higher than density, it will specify the high strength to weight ratio property. As this property is very critical to fabricate, there are many factor should be consider such as the sintering time, temperature, compositions, compaction pressure and other factor. For other factor, studied and research had been done except for compaction pressure. Thus, this project will be focus on the effect of compaction pressure on MWCNTs reinforced Al composites by its mechanical properties, such as density, hardness and tensile strength. Proper experiment and test to study the effect of compaction pressure on this composite will complete the success of the carbon nanotube usage in future. This study is intended specifically on the effect of compaction pressure on final properties of the composite produce. Apart from that, it would also discuss on the microsturucture of the composite fabricated. This report covered the basic literature reviews on the details of the MWCNTs, Aluminium Matrix Composite (AMCs) and related works such as from journals, book, and other references. Other than that, this report covers the methodology and results of the pure Aluminum compaction. Results show that density, hardness and correlation tensile strength of MWCNTs reinforced Aluminum is increase as the effect of compaction pressure. This show that compaction pressure makes the distribution of MWCNTs in matrix more uniform effectively inhibits deformation and produces strengthening effect. The highest compaction pressure use in this experiment, 500MPa has the best hardness value and density which will contribute to fabricate the strength to weight ratio composite material. In summary, the objectives of this experiment have been met. However, there are still a lot of improvements that can be made. These are further discussed in the recommendation part.

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LIST OF ABBREVIATIONS

FYP	Final Year Project
MWCNTs	Multiwall Carbon Nanotubes
Al	Aluminum
PM	Powder metallurgy
MMCs	Metal Matrix Composites
CNT-MMCs	Carbon Nanotubes-Metal Matrix Composites
MWCNTs-Al	Multiwall Carbon Nanotubes reinforced Aluminum composite
FESEM	Field Emission Scanning Electron Micrograph
SEM	Scanning Electron Micrograph
PSA	Particle size analysis
EDX	Energy Dispertion X-ray
XRD	X-Ray Diffration

CHAPTER 1
INTRODUCTION

1.1 Background of Study

Metal matrix composite, specifically on Aluminium matrix composite nowadays experience high demand in automotive and aerospace industry due to its light weight and many other advantages. However, in improving the strength and hardness of Aluminum matrix composite, many research done to reinforcing Aluminium by Carbon nanotubes due to its very low density but high strength and Young's Modulus.

1.1.1 Metal Matrix Composites (MMCs)

Metal matrix composite (MMC) material is a heterogeneous mixture of two or more materials [1], which have been bonded together at a scale that is sufficiently in resulting the consideration a material with properties of its own. The materials used as a matrix in these composites are usually Al, magnesium, copper, titanium, Al-lithium, and super alloys [2]. MMC contains a continuous metallic matrix, and a reinforcement which represent at least a few percent of the material. Metal matrix composites can offer exceptional properties if their reinforcements are good and strong.

1.1.2 Aluminium (Al)

Aluminum (Al) has very low density. It has 3 times weight advantage over iron, nickel and copper. Plus, Aluminum is excellent in conductivity, both electrical and thermal. Usage of Al in its pure form is not applicable due to the main factor, which is low strength. That is why different Al based alloys and composites have been developed to enhance these factors while keeping most of the desired properties of the base metal (Al) present in the form of alloy or composite. Various types of Al composites have been used extensively over the past decades in aerospace and automotive industries. Thus, Aluminum Matrix Composite (AMCs) is fabricated and use widely. Some advantages of AMCs compared to unreinforced materials are as follows [3]:

- Greater strength
- Improved stiffness
- Reduced density (weight)
- Improved high temperature properties
- Controlled thermal expansion coefficient
- Thermal/heat management
- Enhanced and tailored electrical performance
- Improved abrasion and wear resistance
- Control of mass (especially in reciprocating applications)
- Improved damping capabilities.

1.1.3 Carbon Nanotubes (CNTs)

In general, carbon nanotubes had been discovered 30 years earlier, but had not been fully appreciated at that time. Historically, in the late 1950s, Roger Bacon at Union Carbide [4]. found a strange new carbon fibre while studying carbon under conditions near its triple point. He observed straight, hollow tubes of carbon that appeared to consist in graphitic layers of carbon separated by the same spacing as the planar layers of graphite. In the 1970s, Morinobu Endo [5] observed these tubes again, produced by a gas-phase process. Indeed, he even observed some tubes consisting in only a single layer of rolled-up graphite.

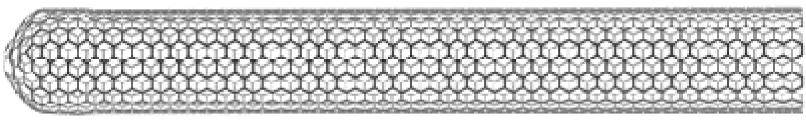

Figure 1: Buckytube or carbon nanotube

In 1991, after the discovery and verification, Sumio Iijima [6] from NEC Corporation observed strange structure of carbon tubes concentric inside each other which are now known as Multi-Wall Carbon Nanotubes (MWCNT) as shown in Figure 2.

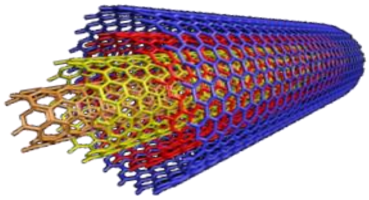

Figure 2: Multiwall carbon nanotubes structure

Nanotubes have molecular dimensions, and consist of perfect graphite sheets rolled into hollow cylinders. There are two types: single-walled with diameters of about 0.5–2 nanometres and multi-walled which have diameters of 2–50 nm. In many respects they resemble the polymer chains used as composite matrices, both have covalently bonded structures, similar dimensions and mechanical flexibility. This makes nanotubes entirely different from traditional fibres such as carbon or glass, which are relatively large with diameters on the scale of micrometres and brittle. The basic mechanical properties such as strength of nanotubes greatly exceed those of other fibres, yet this strength is combined with a low mass density, making them extremely light. Furthermore, nanotubes can conduct heat and electricity down their long axes as efficiently as metals [7].

1.1.4 Multi Wall Carbon Nanotubes reinforced Metal Matrix Composites (MWCNT-MMCs)

As MWCNTs have very low density and extremely high mechanical and electrical properties, it is very suitable and attractive for use as reinforcement in composite materials. MWCNTs reinforced Aluminum will lead to improvement in mechanical properties, especially in Young's modulus and yield strength [8]. Some of criteria should be considered in reinforcing MWCNTs with AMCs are; weight percent composition or concentration of MWCNTs and Aluminium [9], fabrication techniques, mixing period, compaction pressure, sintering atmosphere, sintering temperature and sintering time. If those criteria achieve the requirement, mostly, the yield stress (σ_y) [10] and the maximum strength (σ_{max}) will be increase [11].

In short, using MWCNT in MMC manufacturing is very promising because this could be the way for producing lightweight, ultra high strength, and stiff products made out of Metal Matrix-Nanotubes. Morelli [12] emphasizes that there are two major problems that face scientists and researchers in manufacturing CNT reinforced composites which are: achieving a homogeneous and uniform dispersion of CNT in the matrix, and forming a strong bond at the CNT-metal interface.

1.1.5 Powder Metallurgy (PM)

Powder Metallurgy is a highly evolved method of manufacturing dependable net shaped components. The basic PM production stages are as follows: (1) powder production and preparation, (2) powder blending, (3) powder compaction, and (4) sintering of the compact. These steps are then followed by secondary operations like extrusion, forging, rolling, etc., depending on the application and structure of the final product, and some of these operations are just employed to improve the finishing or readjust the final dimensions [13].

The PM process is a unique part fabrication method that is highly cost effective in producing simple or complex parts closer to final dimensions. Other than that, PM produce complex shapes to very close dimensional tolerances, with minimum scrap loss and fewer secondary machining operations. The physical and mechanical properties of components can be tailored through close control of starting materials and process parameters. Particular properties can be improved through secondary processing operations such as heat treating and cold/hot forming by using this PM. [14]:

1.2 Problem Statement

In aerospace and automotive industry specifically, the challenge is to fabricate material which have high strength to weight ratio composite [15]. As this property is very critical to fabricate, this project will be focus on the effect of compaction pressure on CNTs reinforced Aluminium composites by its mechanical properties, such as density, hardness and tensile strength. When the hardness and tensile strength obtained much higher than density, it will specify the high strength to weight ratio property. Thus, the best compaction pressure can be use to fabricate the demand material use in industry.

1.3 Objectives and Scope of Study

The objectives of the project:

i. To fabricate composites of multi wall carbon nanotube (MWCNTs) reinforced by Aluminum using powder metallurgy technique.

ii. To find out the effect of compaction pressure on the mechanical properties of multi wall carbon nanotube (MWCNTs) reinforced by Aluminum composites.

The scope of study:

The scope of the project includes testing on the mechanical properties of the composites by performing microstructure characterization of the composites, determine; hardness, shrinkage or expansion, density and strength.

Relevancy of the Project

This project is relevant to Mechanical Engineering academic syllabus of Universiti Teknologi PETRONAS (UTP). It incorporates knowledge in Material Science and Engineering, engineering materials, manufacturing technology, solid mechanics, and technical specialization in Material. In addition, it also enhances project management and communication skills.

Feasibility of the Project within the Scope and Time Frame

For this project, the first semester will cover the production of experimental specimens and sintering. The second semester will be concentrated on detail testing of the effect on compaction pressure. Based on the draft methodology, the project's objectives are considered achievable within the given time frame.

CHAPTER 2
LITERATURE REVIEW

2.1 Experimental consideration

The literature on MWCNTs-Al composites is limited to a few numbers of available papers. There have been a lot of trials to produce MWCNTs-Al composites using different types and sizes of CNT synthesized using the different techniques. Three mixing techniques can been used for MWCNTs-Al composites; first, the CNTs also can be mixed with the metallic powder, for example by ball milling, turbula mixing, or sonication, and then the resulting mixture powder is processed into bulk material using PM techniques. Second, the matrix metal can be deposited on the CNT directly forming the composite. Third, the mixture powder could be deposited on a substrate and after the deposition is complete the substrate is removed (the plasma spray forming process). The first processing technique happens to be the most widely researched and is the one used in this study. But, there are some consideration should be focus to fabricate the best MWCNTs-Al composites.

2.1.1 Weight percent composition

C.F. Deng et al had fabricated MWCNTs-Al composites using isostatic pressing followed by hot extrusion. Author said that the nanotubes content affects significantly mechanical properties of composite. The experiment results showed that nanotubes are homogeneously distributed in the composite. Meanwhile, the author examine that 1.0wt% nanotube/2024Al composite is found to exhibit the highest tensile strength and Young's Modulus. The maximal increments of tensile strength and Young's Modulus of the composite, compared with the 2024Al matrix, are 35.7% and 41.3% respectively [16].

Refering to R. Perez. Bustamante, Novel Al-based nanocomposites reinforced with multi-walled carbon nanotubes were produced by mechanical milling followed by pressure-less sintering at 823K under vacuum. The interface between Al matrix and the multi-walled carbon nanotubes was examined using transmission electron microscopy Different nanocomposite compositions were studied, namely trough MWCNTs additions of 0.25, 0.50 and 0.75 wt.%. For comparison, pure Al was also investigated. The author results that values for σ_y and σ_{max} increase as the volume fraction of multi-walled carbon nanotubes increases [9].

Thus, we can conclude that the weight percent and composition of MWCNTs-Al composites must be determine and calculate properly because it gives effect on the final properties of the composite.

2.1.2 Milling time

In a recent study the authors Choi et. al., focused their efforts on emphasizing the effect of CNTs and the variation of grain size on the mechanical properties of Al-CNT composites. Pure Al, and 4 vol.% Al-CNT samples were milled for 6 and 12 hours in a ball mill with BPR of 15:1. The resulting samples were then sealed in a copper can under vacuum, compacted, sintered at 470^{o}C, and then hot extruded with an extrusion ratio of 15:1. The author claims that the grain size calculated from TEM images for pure Al powder was found to be 200nm after 6 hrs of milling which was reduced to 70nm after 12 hrs of milling, and that the grain size of the composite powder was identical to that of the pure Al at the same milling times. After extrusion the grain size was found to be almost unchanged for both the pure and composite samples. Compression testing was conducted for pure and composite samples; the results are shown in table below.

Milling time (hrs)	**Tensile Strength for Pure Al sample**	**Tensile Strength for MWCNTs-Al sample**
6	238	380
12	383	405

Table 1: Compression test results for pure and composite samples [17]

The author didn't comment on the compression results but it is clear from these results that there is a remarkable effect when CNT is added in case of the 6 hrs milled sample. As for the 12 hrs milled samples, the very small grain size and the heavily strain hardened material made the strengthening effect of CNTs unremarkable [17].

Other than that, R. Perez Bustamante et al. use Al powder (99.9% pure, −325 mesh in size) and MWCNTs to produce Al-based nanocomposites. The MWCNTs used in this work were produced by the spray pyrolysis method. Different nanocomposite compositions were studied, and each mixture was blended in an ultrasonic bath for 5 min and mechanically milled in a high-energy shaker mill (SPEX-8000M) using different milling times, 1 hour and 2 hours. Argon was used as the inert milling atmosphere. The weight of the samples was set to 5 g, and the milling media-to-powder weight ratio was 5:1. All milling runs were performed with no addition of processing control agents. Consolidated products were obtained by uniaxial load pressing during two minutes at 950MPa. Compacted samples were pressure-less sintered during 3 h at 823K under vacuum (∼2 Torr).

The pure Al reference sample was not milled; it was only consolidated and sintered at the same conditions. Results are as shown below:

Yield strength, maximum strength and Vickers hardness values obtained in pure aluminum and aluminum-based nanocomposites

Milling time [h]	MWCNT concentration [wt.%]	σ_y [kg/mm^2]	σ_{max} at $\varepsilon = 0.1$ [kg/mm^2]
0*	0.00	6.29 ± *0.3*	8.50 ± 0.5
1	0.00	12.61 ± 0.5	19.85 ± 0.1
	0.25	13.23 ± 0.4	20.63 ± 0.6
	0.50	13.39 ± 0.4	21.70 ± 0.8
	0.75	14.08 ± 1.0	23.27 ± 0.6
2	0.00	13.00 ± 1.1	20.16 ± 1.2
	0.25	15.21 ± 0.8	22.71 ± 1.2
	0.50	19.06 ± 0.9	26.70 ± 0.7
	0.75	23.05 ± 1.7	33.08 ± 1.5

* Reference sample.

Table 2: Comparison of different milling time and MWCNT concentration [9]

Author shown that the effect of milling in two ways: (i) the σ_y value (13 kg/mm2) of pure Al, milled and sintered, compared with the σ_y value (6 kg/mm2) for the reference sample (pure Al not milled and sintered) is increased by more than 100%, and (ii) the overall yield strength is enhanced as the milling time increases. In addition, it is evident that the presence of MWCNTs leads to an increase of the nanocomposite's yield strength [9].

2.1.3 Sintering temperature

L.Ci et al. emphasized that the problem encounter when using temperatures higher than the melting temperatures of Al lies in the severe reactions which may affect the mechanical properties of CNT. It was concluded based on the above results that powder metallurgy involving temperatures lower than the melting point of Al might be the best technique of choice to manufacture Al-CNT composites. Also, it could be inferred that using amorphous MWCNT with carbon deposited on the outer layer might be the best choice when preparing such composites because the defects and carbon will encourage a reaction to take place at the interfacial region [18].

At high temperatures, a reaction may occur in CNT reinforced Al between the Al and the carbon to form Al3C4 on the interface taking the shape of needles which reduce the composite strength. Xu et. al., reported the presence of some Al carbides in an Al-CNT composite manufactured by hot pressing. Also, Zhang et. al., and other researchers observed the formation of Al carbides when the Al-CNT composite was subjected to 800ºC for one hour [9, 19, 20, 21, 22].

2.1.4 Sintering atmosphere

Several Al alloys were made using air atomised powder and conventional press-and-sinter powder metallurgy techniques. These were sintered under nitrogen with a controlled water content which varied from 3 to 630 ppm (a dew point of -69 to -25^{o}C), nitrogen–5%hydrogen, argon and argon–5%hydrogen, all at atmospheric pressure, or a vacuum of <10^{-2} torr. Result gives the best atmosphere for sintering Aluminum in decreasing order is; Nitrogen, Vacuum and Argon [23].

2.1.5 Compaction pressure

To choose the suitable compaction pressure was not an easy process. There were no references discussing the compaction pressure of MWCNTs-Al composites. The closest hit was Al alloys compaction pressures. The Compilation of ASTM Standard Definitions introduced a curve showing the compaction pressure versus the green density, % theoretical of a 601 AB Al alloy [24]. Based on this curve powder compaction pressure of 300, 400, 475, and 550MPa were examined.

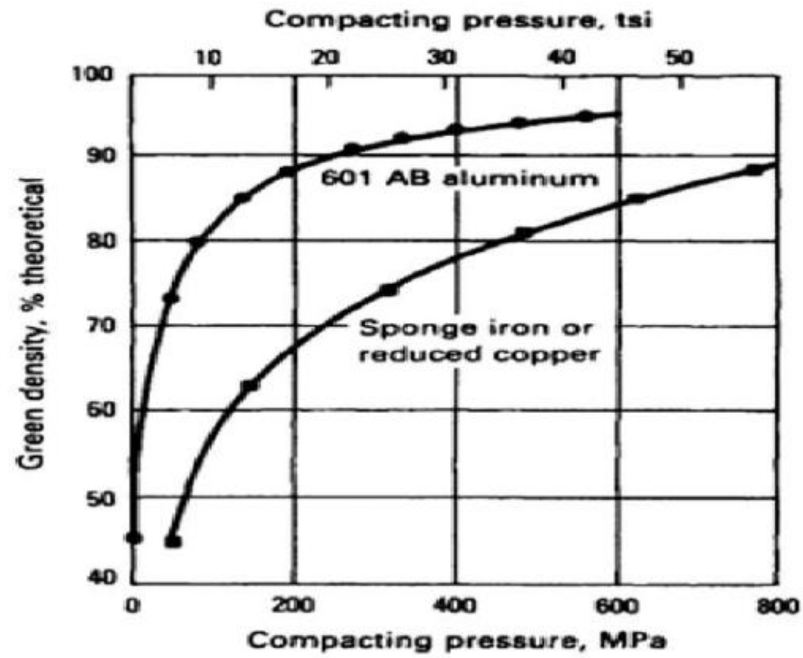

Figure 3: Relationship between green density and compaction pressure of Al alloy [24]

CHAPTER 3
METHODOLOGY

3.1 Project activities

The methodology is formulated based on normal powder metallurgy production and testing. Summary of methodology is shown in Figure 4 below.

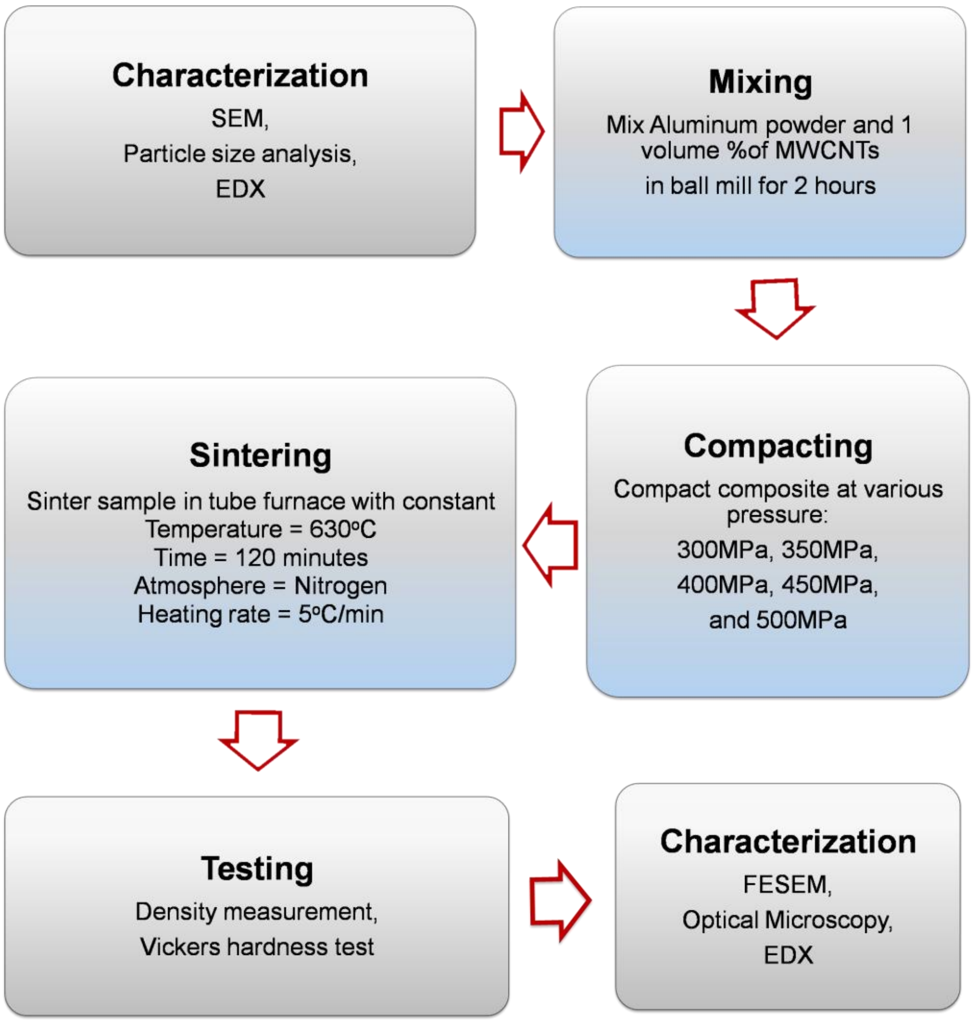

Figure 4: Flow chart of project methodology

The key milestone and datelines for each activity are specified in the Project Gantt Chart in appendices.

3.2 Procedure Identification

3.2.1 Materials composition

The characterization of raw material which is MWCNTs powder and Al powder is done includes particle size analysis, review on particle shape, length and dimensions by Scanning Electron Microscope (SEM) machine. This SEM is done to get the exact picture of the material used by imaging at a magnification of backscattered electron images. Other than that, Energy Dispersive X-ray spectroscopy (EDX) is done for semi-quantitative to determine the ratio between the elements present.

After finish all the steps and specific planning has been done, the material will be fabricated by mixing in ball mill. In reference to R.P'erez-Bustamante et al [9], milling time and the concentration of MWCNTs have an important effect on the mechanical properties such as yield stress and tensile strength of the composites. The composition is calculated by:

1. Determination of theoretical density:

$$\rho_{th} = (\rho_{Al} \text{ x } \%vol_{Al}) + (\rho_{CNT} \text{ x } \%vol_{CNT})$$

2. Volume of the Sample (fixed volume: Ø13mm x 3mm)

$$V = \pi r^2 \text{ x thickness}$$

3. Determination of mass usage by volume percentage:

$$M = \rho_{th} (V)$$

$$m_{Al} = \%vol_{Al} \text{ x } M$$

$$m_{CNT} = \%vol_{CNT} \text{ x } M$$

Thus, the volume percent used is 1% volume of MWCNTs. Then, the MWCNTs and Al is mixed in ball mill for 2 hours. Pure Aluminum sample is also fabricated to be the reference point and comparison purpose.

3.2.2 Compacting

Various compaction pressure is use to study the effect of compaction on the final properties of CNTs reinforced with Aluminium. By H. Abdoli et al [10], the study of densification of nanostructured composite powder, results show that the yield strength is dependable of compacts composites. There are 5 different compaction pressure used, which are 300, 350, 400, 450 and 500Mpa. For each pressure, 3 samples are fabricated. Minimum of 30 samples for this experiment are fabricated. The average value and standard deviation of measured dimension, weight and density is taken.

3.2.3 Sintering

Then, sintering takes place. The sintering is effected by sintering time, atmosphere, temperature and heating rate. Based on G.B.Schaffer et al [23], the best atmosphere for sintering Aluminum in decreasing order is; Nitrogen, Vacuum and Argon. And by R.A Saravanan et a [25], the temperatures higher than 850°C, nitrogen decreases the surface tension of pure Aluminum. The effect is associated to the reaction between Al and nitrogen to form AlN that occurs at temperatures above 850°C.

Thus, for this experiment, nitrogen atmosphere is used. Other specifications used for sintering are, heating temperature of $630^{O}C$ in tube furnace for 120 minutes with heating rate of $5^{O}C$/min.

3.2.4 Preparation for characterization and testing

The surface of the specimens has to be grinded and polished to be ready for testing and this is done by the sample preparation technique. This technique was used in order to obtain sound polished and etched specimens. The same specimens used for the density measurements are then used in this process. Specimens are grinded directly using ascending grinding steps with grit sizes as follows: 180, 240, 320, 400, 600, 800, and 1200. After the samples are ground they are subjected to polishing using alumina solution of 0.5 followed by 0.3 particle sizes. Finally, the samples are etched using (95ml distilled water, 2gms NaOH, and 4gms Na_2CO_3) for 1 min.

3.2.5 Characterizing

Characterization is done; specifically by using Optical Microscopy for 5X, 50X, 100X, and 150X magnification scale. For FESEM, 3k, 5k, 20k, 30k and up to 100k magnification scales are use to examine the microstructure of the samples. Then, X-ray diffraction (XRD) is used for phase analysis. XRD tests are employed for both bulk and powder samples.

3.2.6 Testing

Hardness test is done to ensure the changes and improvement occur effect of the compaction pressure. The measurements were performed at room temperature, at 300gf of load and 15 seconds of dwell time. All readings were taken in Vickers Hardness unit, HV. Five different indentations are made at different sites on each of the samples. Average and standard deviation value are plotted in graphs.

Other than that, correlation of hardness to tensile strength is calculated. This is because; the sample shape produced in this experiment which is small round tablet is inadequate to test for tensile strength by tensile test. In calculating the tensile strength, Vickers hardness (HV) value which obtained from hardness test must be converted into Brinell hardness (HB) and the formula below is use:

Tensile strength = HB x 3.45 (Mpa)

3.3 Tools/ Equipment used

1. Ball mill machine
2. Auto pallet Press Machine
3. Tube Furnace
4. Vickers Hardness Test
5. For characterization:
    a. Scanning Electron Microscope (SEM)
    b. Field Emission Scanning Electron Microscopy (FESEM)
    c. Energy Diffraction X-ray (EDX)
    d. Particle Size Analysis (PSA)

e. X-Ray Diffraction (XRD)

CHAPTER 4
RESULTS AND DISCUSSION

4.1 Pure Aluminum

4.1.1 Scanning Electron Microscope (SEM)

During SEM for Aluminum powder, we have used the magnification of 500, 3000, and 5000 to have the clear image of this powder. It shows irregular size and shapes of the Aluminum powder. The SEM for Aluminum powder:

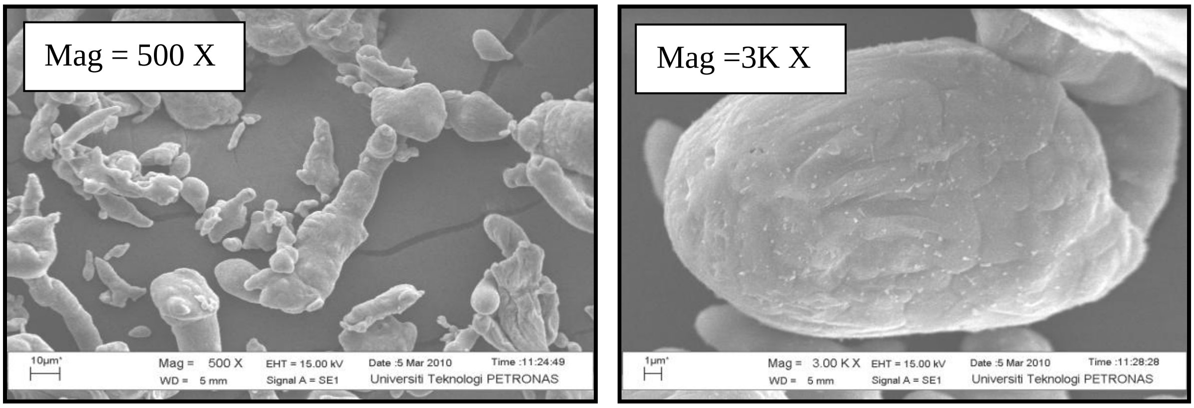

Figure 5: SEM of Aluminum powder

4.1.2 Particle Size Analysis (PSA)

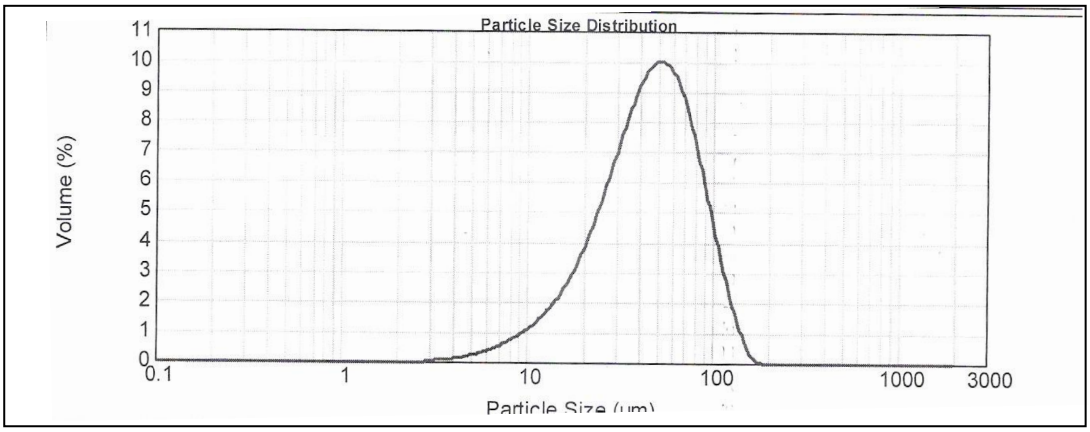

Figure 6: PSA results for Aluminum powder

From this PSA, we obtain the size for aluminium powder is, d (0.5) = 45.739 µm.

4.1.3 Energy Difraction X-ray (EDX)

EDX is done for Aluminum powder because it is formation of Aluminum alloy, which forms percolating network and is termed as matrix phase. The other constituent is embedded in this Aluminium alloy matrix and serves as reinforcement, which is usually non-metallic and commonly ceramic such as Silicon Carbide, SiC and Alumina, Al_2O_3.

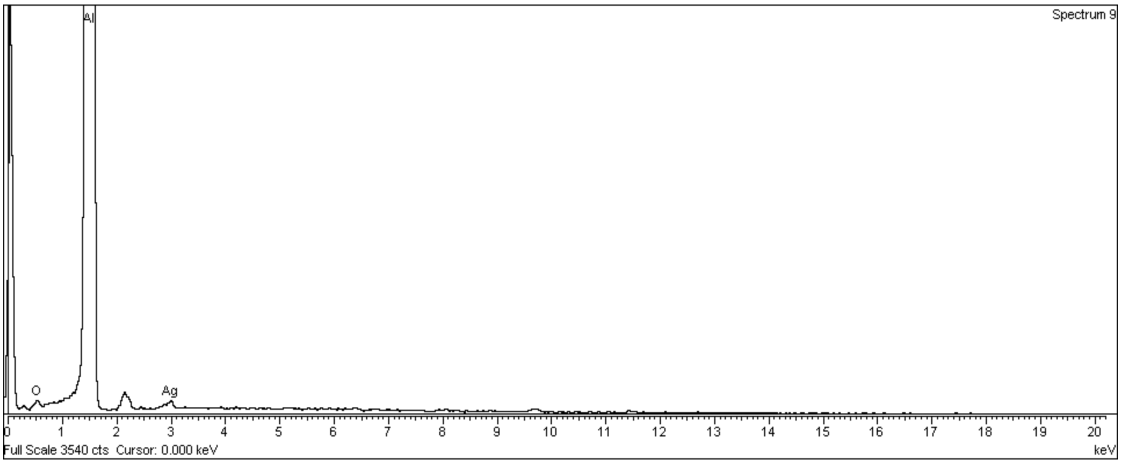

Figure 7: EDX graph for Aluminum powder

The details and percentage of constituent in Aluminum powder is also determined by the EDX method. The results shown below:

Element	**Weight %**	**Atomic %**
O	2.68	4.46
Al	96.43	95.32
Ag	0.90	0.22
Totals	100.00	

Table 3: EDX details for Aluminum powder

4.1.4 X-Ray Diffraction (XRD)

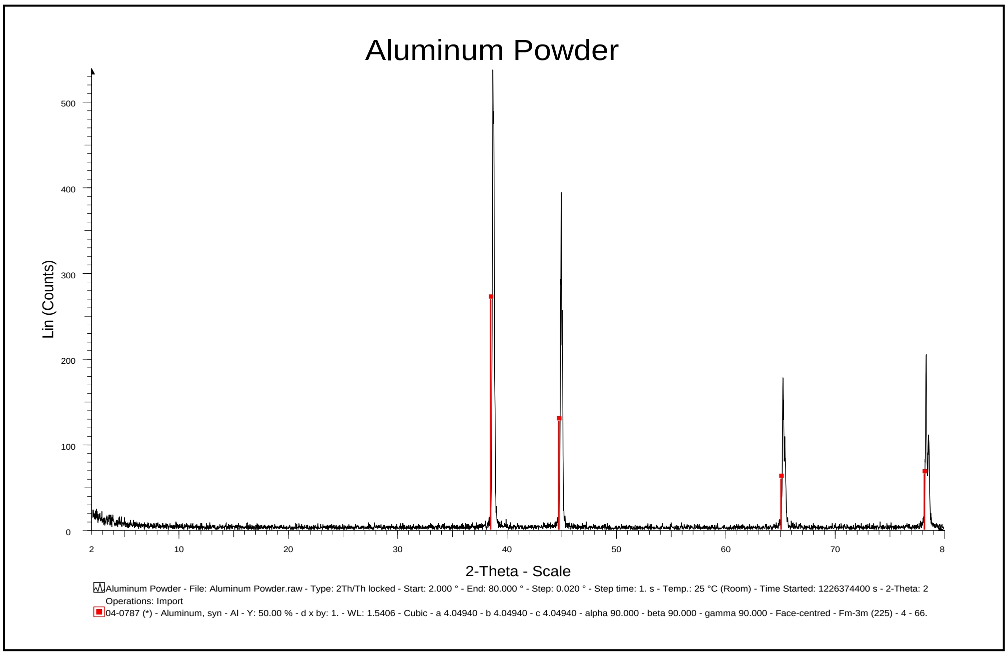

Figure 8: XRD result for Aluminum powder

Thus, by doing this X-Ray Diffraction (XRD), it is known that the Aluminum used is not oxidized. It is important to use non-oxidize Aluminum because Aluminum is easily oxidize and form Alumina, Al_2O_3. If we use the oxidize Aluminum, the final result to determine the effect of compaction pressure on final properties of MWCNTs reinforced Al composite will be effected.

4.2 Multiwall Carbon Nanotubes (MWCNTs)

4.2.1 Scanning Electron Microscope (SEM)

Same as Aluminum, we have done the same techniques of SEM for MWCNTs. The magnification use are; 500, 1000 and 3000. The MWCNTs size is larger than Aluminum powder. Thus, the magnification use is less. These are the results:

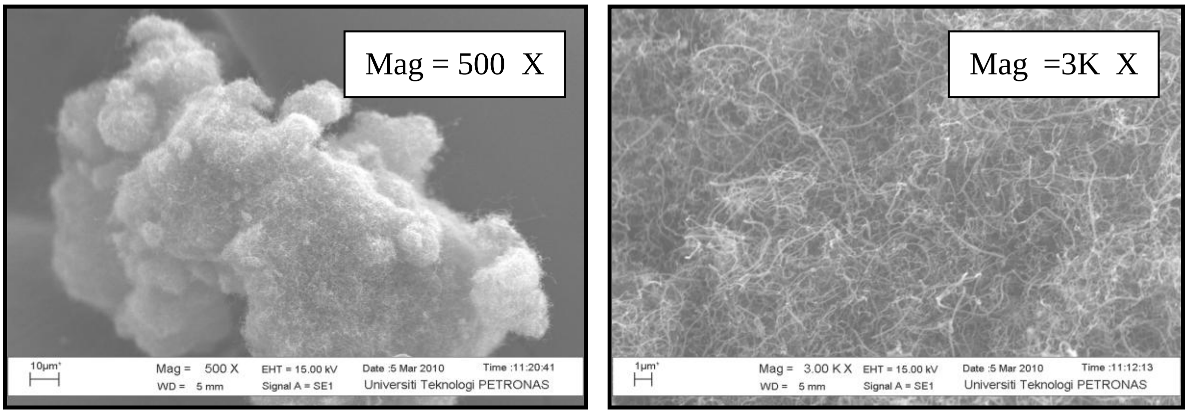

Figure 9: SEM of MWCNTs

4.2.2 Energy Difraction X-ray (EDX)

EDX is done for MWCNTs powder to ensure if there is other materials or contaminant exist in the materials use which might effect the overall experiment results.

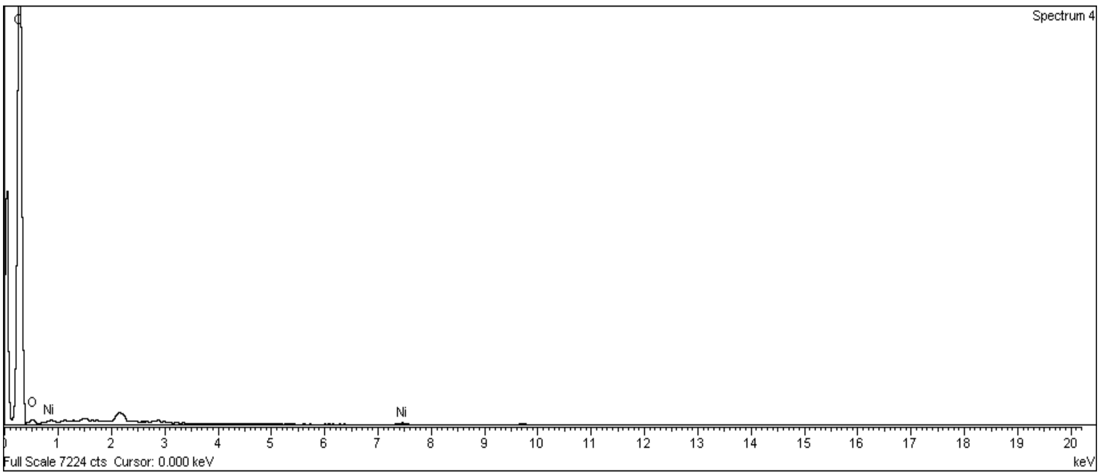

Figure 10: EDX graph for MWCNTs

The details and percentage of constituent in MWCNTs powder is also determined by the EDX method. The results shown below:

Element	**Weight %**	**Atomic %**
C	93.22	95.03
O	6.39	4.89
Ni	0.39	0.08
Totals	100.00	

Table 4: EDX details for MWCNTs

From EDX result, the constituent of MWCNTs use consist of more than 90% Carbon, C which is up to 93.22 weight percent. The other element exists but in only less weight and atomic percent of overall MWCNTs are Oxygen, O and Nitrogen, Ni.

4.3 Multiwall Carbon Nanotubes reinforced Aluminum Composite

There are 5 different compaction pressure used, which are 300, 350, 400, 450 and 500Mpa. For each pressure, 3 samples are fabricated and the average value is taken. Minimum of 30 samples for this experiment are fabricated. The results are compiled in table below.

Pressure	Density (g/cm^3)		Hardness (HV)		Hardness correlation to Tensile Strength (M Pa)	
	Pure Al	CNT 1%	Pure Al	CNT 1%	Pure Al	CNT 1%
300	2.560	2.42	29.3	29.8	71.7	72.9
350	2.579	2.48	30.9	31.0	75.9	77.8
400	2.554	2.52	31.3	31.8	78.5	79.7
450	2.586	2.56	31.6	33.5	79.3	83.6
500	2.597	2.59	31.8	35.9	79.8	89.4

Table 5: Effect of compaction pressure on density, hardness and tensile strength

Result shows that the density, hardness and tensile strength are increase with the increase of compaction pressure, both for pure Aluminum and MWCNT-Al composite. The graph and analysis of each property are discussed.

4.3.1 Density

Due to different density value for composite and pure Aluminum (2.7 g/cm^3) the composite density of 1% volume of MWCNTs can be calculated as below:

$$\rho_c = V_m\rho_m + V_f\rho_f = (0.99)(2.7) + (0.01)(1.8) = 2.691 g/cm^3$$

The composite density is lower than the density of pure Aluminum because MWCNTs have ultra low density, which is 1.8 g/cm3. Comparison for composite and pure Aluminum density is done and shown below.

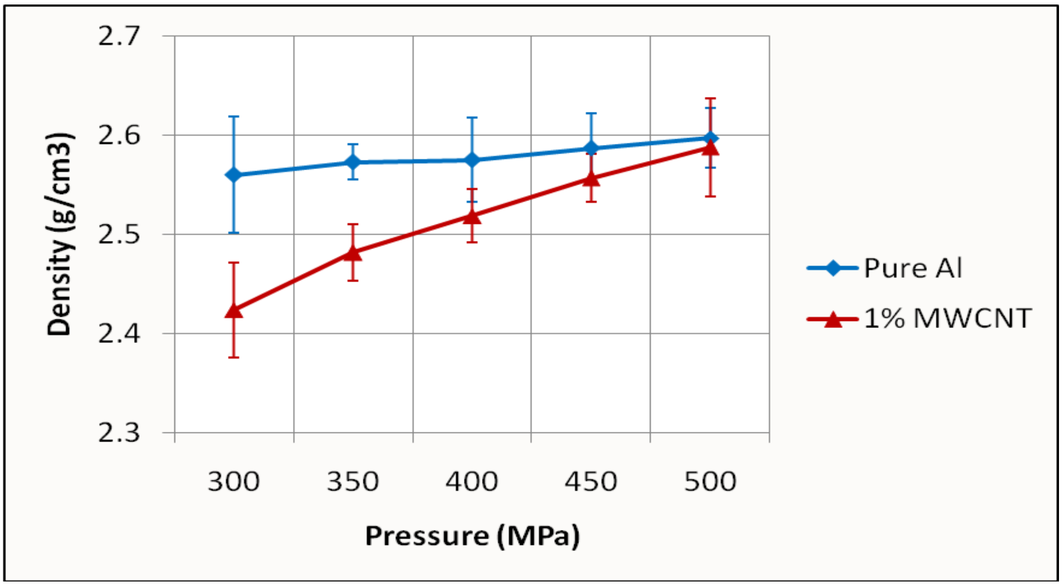

Figure 11: Density comparison between Pure Aluminum and MWCNT-Al composite

Result show that both pure Aluminum and composite (MWCNT 1% reinforced Aluminum) density increase when the pressure increase. For pure Aluminum, the increment from 300Mpa to 500Mpa is approximately 1% while for composite is 5%.

However, density for composite is lower than pure Aluminum in a range of 3%. For strong, lightweight component material use in industry such as aerospace where weight is critical, we require a material with the best strength to weight ratio. Thus, this low density of MWCNT 1% reinforced Aluminum composite will contribute to this specific strength. This is the main advantage of the low density of composite compare to the pure Aluminum. MWCNT 1% reinforced Aluminum composite will have better strength to weight ratio compare to the pure Aluminum.

4.3.2 Hardness

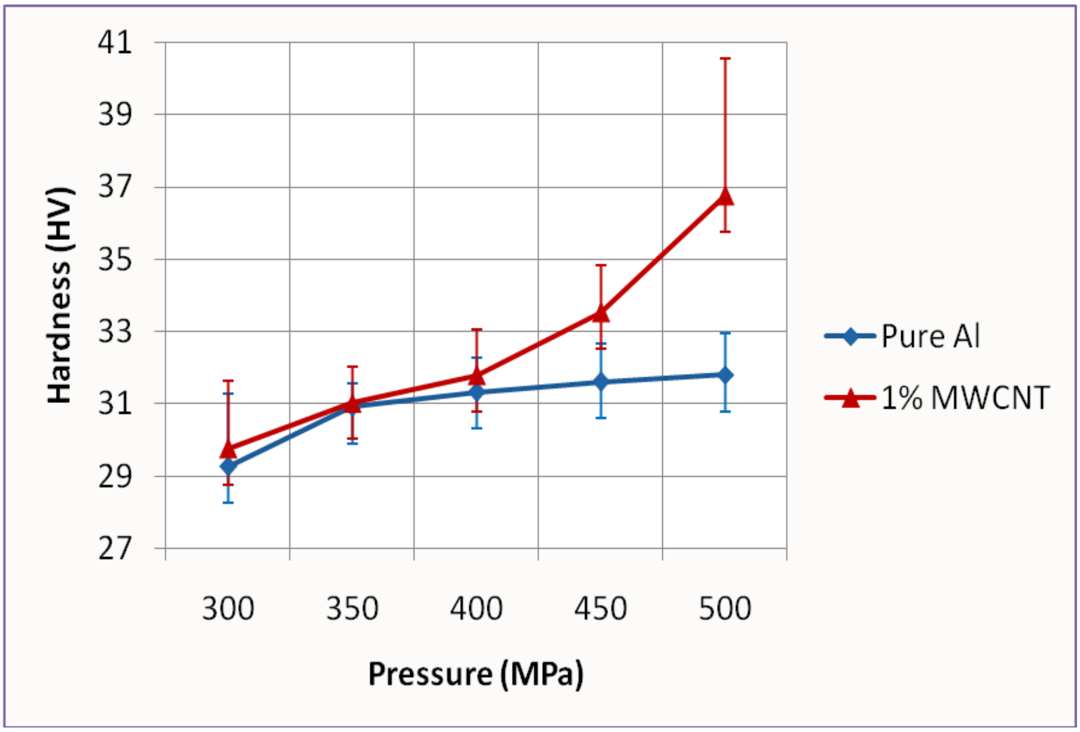

Figure 12: Hardness comparison for Pure Aluminum and MWCNT-Al composite

For hardness, both results in increase of hardness with increasing pressure. The increase is approximately 9% and 20% for pure Aluminum and MWCNT 1% reinforced Aluminum composite. Hardness percentage difference between pure Aluminum and nanocomposite for 500Mpa is about 13%.

This different is due to the usage of 1% Multiwall Carbon Nanotubes (MWCNT). The load apply is transfer to MWCNT from the Aluminum. The higher the pressure use, the reinforcement between MWCNT and Aluminum become better. Thus, higher load transferred to the MWCNT.

4.3.3 Correlation of Hardness to Tensile Strength

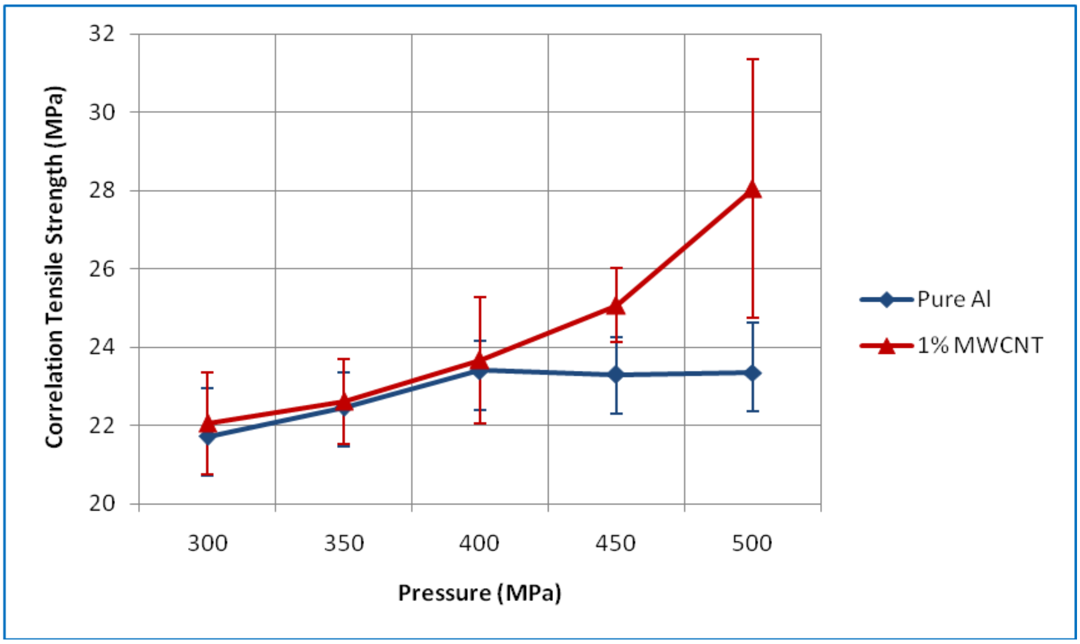

Figure 13: Tensile strength comparison for Pure Al and MWCNT-Al composite

In calculating the tensile strength, Vickers hardness (HV) must be converted into Brinell hardness (HB) and the formula below is use:

Tensile strength = HB x 3.45 (Mpa)

Thus, the result show tensile strength is increase when pressure is increase for both pure Aluminum and nanocomposite. Tensile strength for Aluminum increase for about 11% from 300Mpa to 500Mpa while for MWCNT 1% reinforced Aluminum composite, 23%.

4.3.4 Comparison on density and Hardness

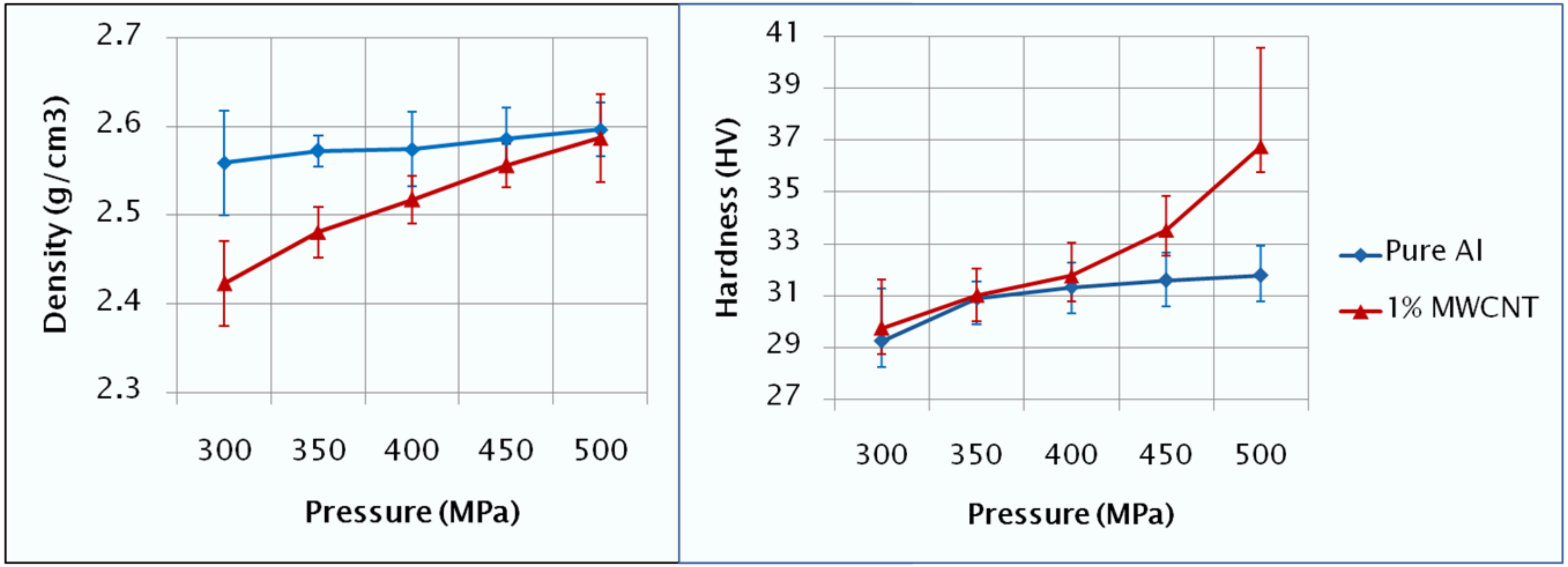

Figure 14: Relation of density and hardness for Pure Al and MWCNT-Al composite

Both density and hardness values are increase as the effect of compaction pressure. When compaction pressure increase, usage of 1% of MWCNTs composite results in lower density but higher hardness compare to pure Aluminum. This result will achieve the required high strength to weight ratio composite.

$$\text{Specific strength} = \frac{\text{Hardness}}{\text{Density}}$$

4.3.5 Analysis on FESEM of MWCNTs reinforced Al

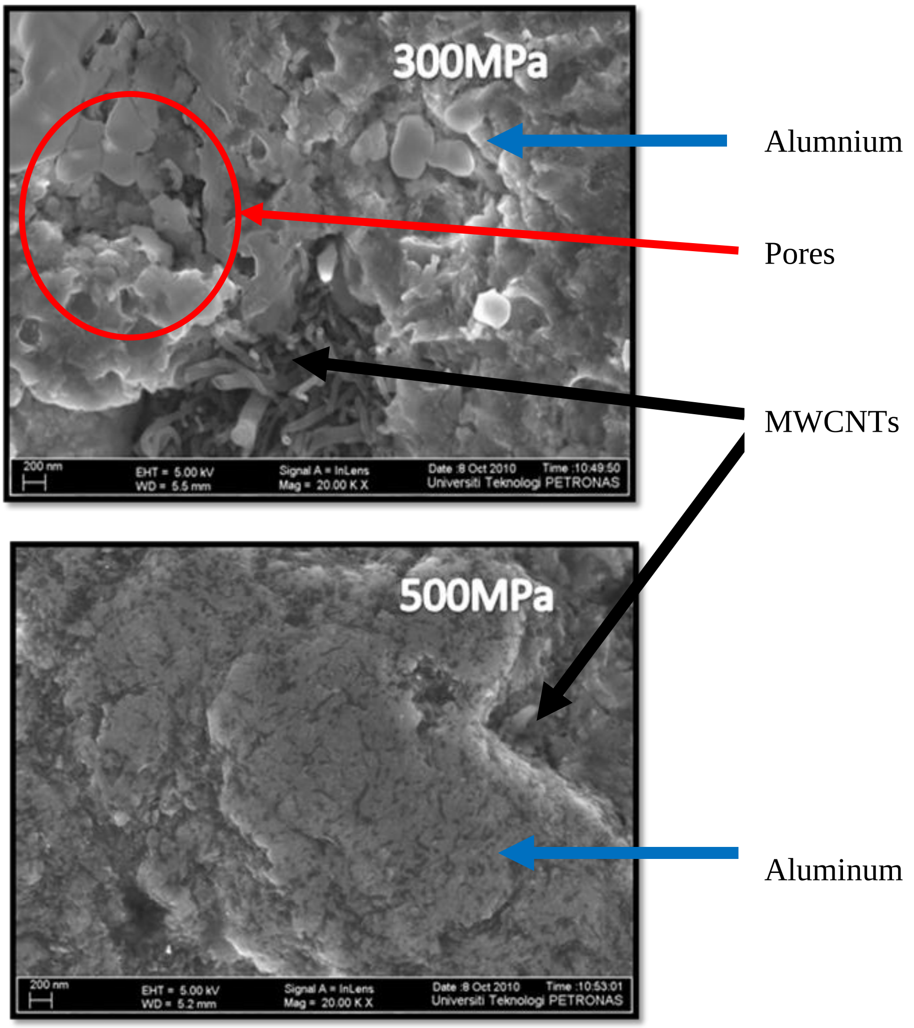

Figure 15: MWCNTs reinforced Al composite at magnification of 20K at compaction pressure of (a) 300MPa and (b) 500MPa

The FESEM is done at the fracture surface. At 300MPa, the bonding between MWCNTs and Aluminum is poor. MWCNTs easily slip away, produces pores [26]. As said by Alain Rochefort, MWCNTs have "Russian doll" structure which only bonded to its constituent tubule neighbours by weak Van der Waals forces. This means, bonding the outer tube of MWCNTs would not anchor the inner tubes. The inner tubes slippage will hurdle strong bond between Aluminum and all the constituent tubules of MWCNTs. Presenting defects in multiwall structure might help to overcome this problem [22]. Thus, compaction pressure can contribute to present this defect, automatically improve the bond, minimize the reinforcement problem and produce high strength to weight ratio composite.

When compaction pressure increases to 500MPa, the MWCNTs and Aluminum distribute homogeneously [16, 27] and form a stronger bonding. Some nanotubes act as bridges across cracks, prevent MWCNTs to slip and successfully inhibits deformation.

4.3.6 Energy Diffraction X-Ray (EDX)

To ensure the results of MWCNTs-Al composite as clarify before is not effected by other factor, EDX is done.

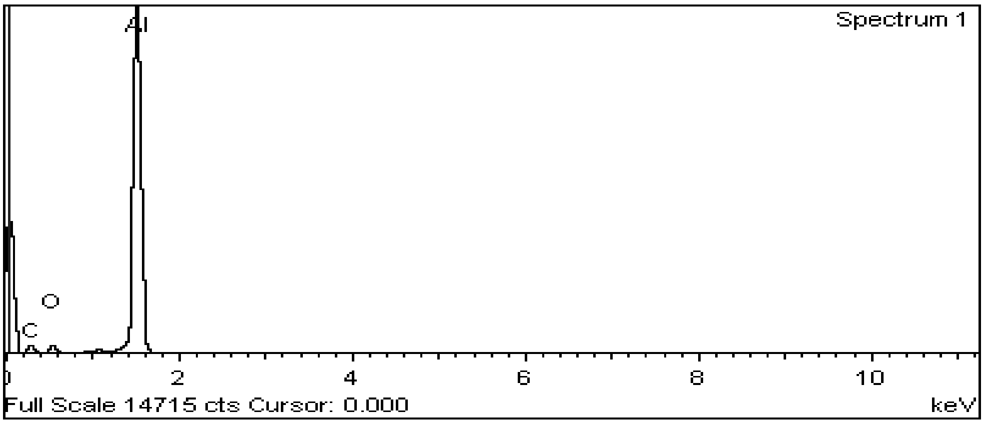

Figure 16: EDX graph for MWCNTs-Al composite

The details and percentage of constituent in MWCNTs powder is also determined by the EDX method. The results shown below:

Element	Weight %		Atomic %	
	300MPa	500MPa	300MPa	500MPa
C	34.35	27.61	61.32	43.70
O	10.97	10.93	12.30	12.99
Al	54.68	61.46	36.37	43.30
Totals	100.00	100.00		

Table 6: EDX details for MWCNTs-Al composite

From EDX result, the constituent of MWCNTs-Al composite mainly consist of Aluminum, Al for about 54 to 62 weight percent while Carbon, C which is up to 27 to 35 weight percent. The other element exists but in only less weight and atomic percent of overall MWCNTs is Oxygen, O.

CHAPTER 5
CONCLUSION AND RECOMMENDATION

5.1 Conclusion

Density, hardness and correlation tensile strength of MWCNTs reinforced Aluminum is increase as the effect of compaction pressure. This show that compaction pressure makes the distribution of MWCNTs in matrix more uniform effectively inhibits deformation and produces strengthening effect.

High compaction pressure, 500MPa will contribute to fabricate the strength to weight ratio composite material

As far as this research is concern, the objectives have been met. However, there are still a lot of improvements that can be made. These are further discussed in the Recomm

5.2 Recommendation

Based on the current results, the bonding between MWCNTs and Al can be improved by usage of wax. When bonding between matrix and fiber is improved, the final properties such as hardness and tensile strength will be improved.

The condition of the MWCNTs should be analyzed further. The compaction pressure might produce defect in the internal structure of MWCNTs to sustain itself from inner tube slippage. Higher magnification of microstructure view can be use.

Other than that, Transmission Electron Microscopy (TEM) can be use to characterize the composite. Even though TEM analysis is very expensive, it is considered to be a valuable characterization tool that provides a wealth of information about the internal structure of MWCNTs-Al composites, and was very helpful in detecting the presence of nanostructures, and any carbides structures.

The compaction pressure can be increased until the limitation or fatigue failure is achieved. Till then, we can exactly use the best compaction pressure for mass production and usage of MWCNTs reinforced Al composite.

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APPENDICES

APPENDIX A: FYP 1 GANTT CHART

Action plan	Week														
	1	2	3	4	5	6	7	Mid semester break	8	9	10	11	12	13	14
Problem definition															
Produce proposal	■	■													
Project planning		■	■												
Literature Review			■	■											
Design															
Produce experimental procedure				■	■										
Produce conceptual assumption				■	■										
Raw material															
Understand the properties composites			■	■	■										
Prepare raw material					■	■	■								
Mixing															
Mixed the composites by forming technique									■	■	■				
Compacting															
Compact composites in various compaction pressure									■	■	■	■	■		
Sintering															
Sinter composites									■	■	■	■	■		
Testing															
Basic test for compaction pressure effect												■	■		
FYP 1															
Submission of Preliminary Report				■											
Submission of progress report									■						
Seminar									■						
Submission of interim report final draft															■
Oral presentation															■

APPENDIX B: FYP 2 GANTT CHART

Action plan	Week														
	1	2	3	4	5	6	7		8	9	10	11	12	13	14
Compacting								Mid semester break							
Compact composites in various compaction pressure	■	■	■	■											
Sintering															
Sinter samples					■	■	■								
Preparation for Testing															
Mounting, grinding and polishing							■		■	■					
Testing															
Basic test for compaction pressure effect										■	■	■			
Characterize Raw material															
FESEM, OM, XRD												■			
FYP 2															
Submission of Progress Report I				■											
Submission of progress report II									■						
Seminar									■						
Poster Exhibition												■			
Submission of dissertation final draft															■
Oral presentation															■
Submission of Dissertation (Hard Bound)															■